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ORIGINAL PAPER

High-density polyethylene pipe with high resistance to slow crack growth prepared via rotation extrusion

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Abstract Slow crack growth (SCG) is one failure principal mode in polyethylene (PE) pressure pipe applications. In the conventional extrusion process, the molecular chains in the plastic pipes are oriented along the axial direction, which are disadvantageous to their resistance to SCG. In order to change the orientation direction of molecules in the plastic pipe, a new rotation extrusion processing system was designed to extrude high-density polyethylene (HDPE) pipes, and a thorough research was done on the effect of the rotation speed on its microstructure and resistance to SCG during the rotation extrusion. The experimental results showed that when the die rotated during the extrusion process of PE pipes, the hoop stress exerted on the polymer melt could make the molecular orientation deviate from the axial direction, and therefore the consequent multi-axial orientation of molecular chains could be obtained. As a result, the PE pipe with better resistance to SCG was prepared. Compared to the PE pipe produced by the conventional extrusion, the crack initiation time of the PE pipe manufactured by the novel method increased from 27 to 57 h.

Keywords Rotation extrusion · High-density polyethylene · Pipe · Slow crack growth · Orientation

Introduction

Since polyethylene (PE) is distinctive for its lightweight, low cost, corrosion resistance and easily processing and welding, as soon as introduced into the pipe production, it has arouse extension concern and has been widely applied to convey

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gas, oil, water, and sewage [1, 2]. When designing a pipeline system, the proper operating lifetime and reliability are crucial factors for the plastic pipe's application. However, PE pipes often fall in a brittle manner after they are long exposed to the service temperature and low stress, involving the formation of a craze at a point of stress concentration and the subsequent growth and fracture of the material [3]. As one of the principal failure modes, this long-term brittle failure, so-called slow crack growth (SCG), limits the practical service life of PE pipes, and then how to improve their resistance to such failure is of great significance [4].

A number of studies have been carried out to understand SCG phenomenon and improve the PE resistance to SCG. Brown measured the rate of SCG as a function of molecular weight and branch density of PE [5, 6]. The results showed that SCG was originated from chain disentanglement in the fibrils. High molecular weight as well as short chain branches could improve the resistance to SCG. Bubeck revealed that increasing the branch length from methyl to hexyl was beneficial to better resistance to SCG [7]. A typical example of improving PE pipe's performance through chemically modifying the molecular structure of PE was the synthesis of PE100 resins with bimodal molar weight distributions, where the low molecular weight presented stiffness and good processability while the high one enhanced the resistance to SCG [8]. Moreover, the final morphology of a polymer product is vital in determining the resistance to SCG. Gent confirmed that molecular orientation had a significant effect on the facture behavior of PE [9]. The tear energy along the hoop direction was higher than that along the axial direction. Faulkner revealed the effect of molecular orientation on environmental stress cracking of ABS in methanol [10]. The results showed that the critical tensile strain at which the crack formed on the surface of the specimen in bending was significantly lower when the tensile stress was perpendicular to the melt flow. Lu measured the lifetimes to failure of PE pipe in both directions parallel and perpendicular to the axial direction [11]. When the molecules were preferentially oriented parallel to the tensile stress direction, they would be more amenable to SCG than if they were perpendicular to the stress.

However, in the conventional extrusion process of plastic pipes, the molecular chains orient along the axial direction [12, 13]. Unfortunately, the internally pressurized pipe is subjected to a hoop stress twice as high as the axial stress in its application [14]. Thus, a crack often propagates through the pipe wall, parallel to the main axis [15]. As molecular orientation in samples has a positive effect on the resistance to SCG, it is expected that molecular orientation in the hoop direction of PE pipes could improve the resistance to SCG.

When a polymer is subjected to external stress, the molecular chains in the melt orient along the direction of the stress [16]. Hence, by controlling the direction of the stress imposed on the melt, various orientation directions of the molecular chains may be available, which in turn facilitates the development of rotation extrusion. During the plastic pipe's extrusion, die or mandrel rotates so that a hoop stress is generated except the axial stress, resulting in during the extrusion process, the direction of the resultant force deviates from the axial stress and a tangential stress field can be provided [17–19]. Therefore, the molecular chains do not orient along the axial direction and by changing the relative magnitude of the axial and hoop stress, the orientation direction can be adjusted, and physical and mechanical properties in the axial and hoop directions of plastic pipe can be balanced [20]. Deberdeev found that the yield and tensile strength of PE pipe in the hoop direction increased with increasing the rotation speed while those along the axial direction decreased slightly [21]. The study of Chung showed that the orientation of the reinforcing fibers in glass fiber filled polypropylene pipe was controlled effectively by rotational extrusion, resulting in the breaking load of 75 N along the hoop direction increased by ~40% compared to the conventional extruded pipe [22]. Jiang rotated mandrel during PE pipe extrusion. The results showed that the highest hoop strength of the pipe by rotational extrusion reached 90 MPa, almost four times of normal strength [23]. Previous research mainly focused on the enhancement of plastic pipe's short-term mechanical strength along the hoop direction, however, the effect of rotation extrusion on its long-term mechanical property, an essential indicator of the PE pipe's high performance, was rarely reported before and its effect on the structure also has not been well investigated up to now.

To understand the relationship between rotation extrusion, morphological structure, and long-term mechanical property of the prepared pipes so as to optimize the processing parameters for the PE pipe with better performance, our research group has designed and manufactured a novel rotation extrusion processing system [24]. This system has the following features: first, both the mandrel and the die can rotate, either separately or integratedly, in the same direction or in the opposite directions; the rotation speed and direction can be easily adjusted by the motor and gear reduction system, so as to change the hoop stress imposed on the melt. Second, the system has a hollow mandrel, through which a cooling media, such as the air and water, can be transmitted to cool the interior surface of the pipe. As a result, its inner and outer wall can be simultaneously cooled down, and their cooling rate and temperature gradient cross the pipe can be adjusted by changing the temperature and flow rate of the cooling medium. With the novel rotation extrusion system, the crystallization and orientation of PE can be even controlled by adjusting the applied hoop stress and cooling rate, and the PE pipes with higher performance can be manufactured [25, 26]. As a part of systematical investigations, this article focused on the effects of the die rotation speed on the structure and the resistance to SCG of the PE pipes obtained via rotation extrusion.

Experimental

Materials

The material used in this work was a commercially available PE80 grade pipe resin TR480, provided by Sinopec JinFei Petrochemical Co. Ltd (Shanghai, China). It had a melt flow rate of 0.14 g/10 min, measured at 190 °C under 2.16 kg. The weight average molecular weight was 450,000 g/mol with a molecular weight distribution (M_w/M_n) of about 6.54. Arkopal OP-10, a detergent, was purchased from Kelong Chemical Agents Co. Ltd (Chengdu, China) to accelerate the SCG tests.

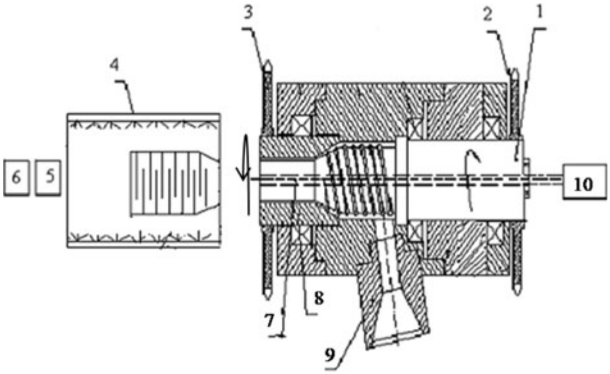


Fig. 1 Schematic diagram of pipe's rotation extrusion equipment [23]. *1* Mandrel, *2* drive system, *3* drive system, *4* vacuum sizing box, *5* traction device, *6* cutting device, *7* flow route of cooling medium, *8* die, *9* adapter block, and *10* inlet of cooling medium

Apparatus and sample preparation

In order to change the molecular orientation in PE pipe, a novel plastic pipe extrusion equipment was designed and manufactured by our research team, the schematic diagram was shown in Fig. 1, and its detailed structure was described elsewhere [24]. In this study, the rotation of the die could be driven by a motor through a series gears; accordingly the hoop stress was imposed on the melt, which could be easily adjusted by the motor and gear reduction system. And, the system had a hollow mandrel that could transmit compressed air to inflate the pipe so as to effectively prevent the pipe's shrinkage during the rotation extrusion and achieve the rotation extrusion. The extruded PE pipe was named as DPE_x, where x was the rotation speed. When the rotation speed of the die was 0, the extruder was equivalent to the PE pipe manufactured by conventional extrusion, named as TPE. The diameter and wall thickness of final high-density polyethylene (HDPE) pipe obtained in this experiment were $\sim$32 and $\sim$3 mm, respectively.

Characterization

Cone tests for SCG

The PE pipe's resistance to SCG was evaluated by an environmental stress crack test called cone test according to ISO 13480: 1997. In this test, a 15 cm long pipe was cut from the prepared PE pipes and inserted by a metallic cone at a constant rate. As the diameter of the metallic cone used in this test was 1.12 times that of the nominal internal diameter of the pipes, the driving force for crack propagation was generated after the cone insertion. Under this condition, a single 10-mm long notch was cut by a fresh razor blade along axial direction at one end of the pipe. Then the assembly was immersed in 5 wt% aqueous solution of Arkopal OP-10 at a well controlled temperature of 80 °C. The crack length increment of the pipes was measured at regular time intervals. Then the curve of the crack length increment versus time was plotted and extrapolated to the time axis, where the intersection point of the curve and time axis was defined as the crack initiation time.

DSC analysis

The thermal analysis of the samples was conducted using a Q20 differential scanning calorimetry apparatus (TA, America), calibrated using indium and zinc standards. The 5–8 mg specimens were heated from 50 to 160 °C with a heating rate of 10 °C/min, under nitrogen flow.

SEM observation

The crystal structure of the PE pipes and fibrils morphology in the fracture surfaces for various pipes after the cone test were observed by using an Inspect F (FEI) SEM instrument at 0.5 Torr and 20 kV. The SEM samples were gold-sputtered prior to observation. In order to investigate the crystal structure, the samples were cut from the pipes and put into the permanganic etchant at 50 °C for 3 h to remove the amorphous phase. The permanganic etchant used was a 1% w/v solution of potassium permanganate in mixed acid composed of three parts concentrated sulfuric acid and one part of concentrated orthophosphoric acid [27]. Then the etched samples were carefully washed by 5 wt% aqueous solution of sulfuric acid, hydrogen peroxide, and distilled water.

Thermal shrinkage

The strips of $\sim$2-cm length were cut along the axial direction from the outer wall of the prepared PE pipe, put into glycerol at 145 °C in order to remove surface constraints when melting the samples [13, 28], and held at that temperature until no further dimensional changes occur. The shrinkage ratio was then measured according to the change of length of the strip before and after heating, which was defined as the ratio of the initial length divided by the final length of the heating sample.

Results and discussion

The effect of the rotation speed on slow crack growth resistance of PE pipe

In PE pressure pipe applications, SCG, is one of the principal failure modes, which is initiated at a defect or stress concentration in the pipe [29]. In this study, the cone test described in ISO 13480: 1997 IDT was used to measure the resistance to SCG of PE pipes. Figure 2 depicted the crack length's increase versus time for the rotation pipes and the conventional pipe in the cone test. It was seen that SCG process was involved into two stages, i.e., the crack initiation stage and the linear steady crack growth stage. The crack initiation time of TPE was 27 h. However, when the die was in rotation, this time could increase rapidly and the crack growth rate was also lower

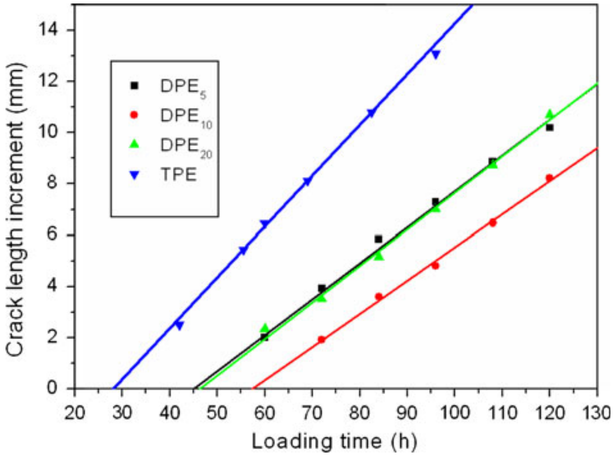


Fig. 2 The crack length's increase versus time for the rotation pipes and the conventional pipe in the cone test

than that of TPE. When the rotation speed was <10 r/min, the crack initiation time reached a maximum value, 57 h, 2.1 times that of TPE. Apparently, the crack initiation time exhibited a nonlinear relationship with increasing rotation speed. When the rotation speed was <10 r/min, the crack initiation time increased with increasing the rotation speed. However, a further increase in the rotation speed led to the decrease in the crack initiation time. However, it still was higher than that of TPE.

After stress cracking of the PE pipes occurred, the first fracture region ahead the notch was examined by SEM, which was helpful to better understand the crack mechanisms. From Fig. 3, it was clear that the fracture morphology of the rotation PE pipe was similar to that of the conventional PE pipe, and the fracture zones were highly fibrillated, which was a typical signal of brittle fracture by SCG. As Fig. 3a clearly showed, TPE had numerous microvoids in the fracture region where the

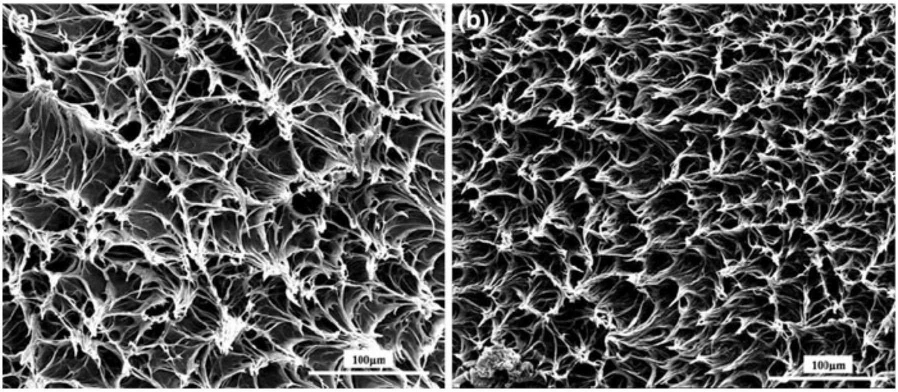


Fig. 3 SEM micrographs of fracture zone in **a** TPE and **b** DPE$_{10}$

fibrils were slender. However, when the die rotated, although the fracture region of DPE was also covered with many microvoids, its fibrils were in clusters and the number of the fibrils was larger than that of TPE. Thus, the fibrils in the rotation pipe became denser and thicker compared to that of the conventional pipe, suggesting the fibrils could withstand the applied stress for a longer time and therefore the rotation pipe had better resistance to SCG. This was consistent with the results of longer crack initiation time.

All those indicated that the rotation of the die could remarkably improve the resistance to SCG, which was resulted from the special morphology by the rotation extrusion.

Effect of rotation speed on the morphology structure of the PE pipe during the rotation extrusion

As a decisive factor to mechanical properties of a polymer product, its final morphology is influenced greatly by the processing conditions such as stress and cooling rate. It is convinced that upon the external stress, the molecular chains in the polymer melt extend along the direction of the stress field. In the conventional extrusion process, the pipe is generally longitudinally drawn. The large tensile stress imposed by the traction device is parallel to the pipe's axis so that the molecular chains in the pipe are expected to be oriented along the axis direction. Figure 4 showed the SEM micrographs of TPE's etched external surface parallel to extrusion direction. It was clear that the external surface of TPE was studded with some more cluster-like crystallites which oriented along the axial direction.

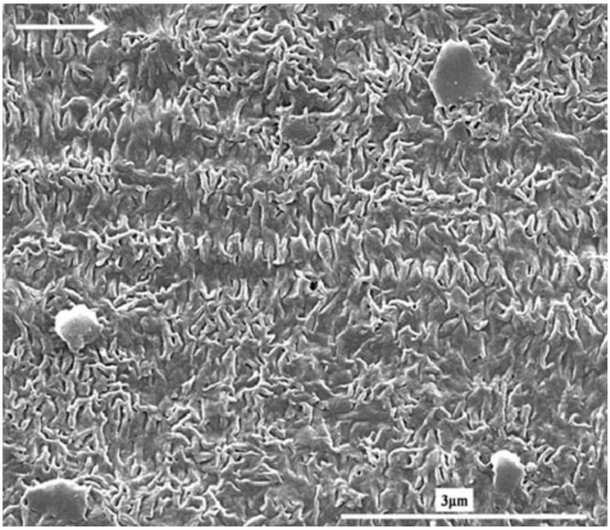


Fig. 4 SEM micrographs of the etched outer surface of the PE pipe produced by the conventional extrusion, 40,000×. The direction of *arrows* showed the extrusion direction

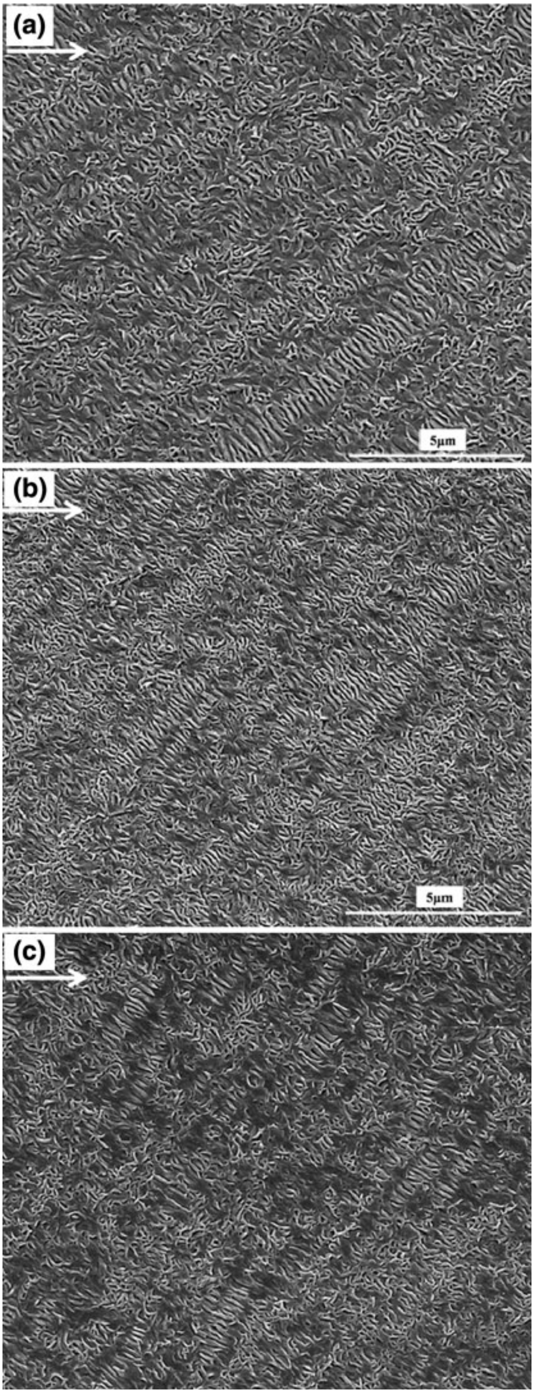
When the die rotated, a macroscopic hoop stress was imposed on the PE melt besides the axial stress so the resultant force was not along the axial direction, bringing changes to the orientation direction of the molecular chains. As shown in Fig. 5, the surface of the rotation pipe were covered with the orientation lamella, which were not oriented along the axial direction but at small angle relative to the extrusion direction and achieving the multi-axial orientation in the PE pipe. In other word, compared with the conventional PE pipe, the orientation along the axial direction in the prepared PE pipe decreased and the orientation along the hoop direction increased. For example, when the die was rotated at 5 r/min, the orientation angle relative to the axial direction was 43°. With the increasing rotation speed of the die, the hoop stress imposed on the melt became bigger so at constant axial force the resultant force leaned to the hoop direction more markedly. As a result, the orientation angle relative to the axial direction became bigger. From Fig. 5, it was clear that when the die was rotated at 20 r/min, the orientation angles relative to the axial direction increased to about 60°, higher than that obtained by rotation at 5 r/min, 43°. Figure 6 represented schematic illustration of the relation between the orientation state and the rotation speed of die. To the best of our knowledge, the orientation angle's change with the rotation speed has not been reported so far.

A melt orientation molecule in a sample tends to become random from the past extending state, usually reducing the sample's length along the orientation direction. Therefore, the change of the sample's length before and after the heating can represent the orientation degree of molecular chains in the PE pipe [13, 28]. When the sample's length decreased after the heating, the shrinkage ratio of the sample was <1, which meant that the molecular orientation existed there. In the conventional extrusion process, the pipe is only subjected to the axial force, so that the molecular chains always tend to orient along the axial direction. Good evidence came from Fig. 7, which showed that TPE's shrinkage ratio along the extrusion direction was 0.29 after the heating. When die rotated, the shrinkage ratio rose, so it could be concluded that the orientation degree along the axial direction decreased. Because when the hoop stress by die rotation was imposed on the melt, the orientation direction of the molecular chains became unparallel to the extrusion direction, which also has been proved by the results of SEM. As a result, the orientation degree along the axial direction decreased and that along the hoop direction increased. After the heating, its shrinkage ratio not only increased, but also was larger than 1.

DSC experiment also can demonstrate the changed crystal structure in the sample. Figure 8 showed DSC curves of the PE pipe by the conventional extrusion and the pipes obtained at the different die rotation speed. As could be seen, all melting peaks shared the almost same shape, but the melting temperature of the PE samples by the rotation extrusion was higher than that of the TPE samples. With the increasing rotation speed, the melting temperature of the samples tended to increase, but at the high rotation speed, it decreased.

During the rotation extrusion process, the PE melt was subjected to the hoop stress induced by die rotation as well as the axial stress. Therefore, the resultant stress was bigger than the sole axial stress in the conventional extrusion, which was favorable to promote the orientation of the molecular chains. As a result, the total

Fig. 5 SEM micrographs of the etched outer surface of the PE pipe obtained at the different rotation speed. **a**, **b**, and **c** was 5, 10, and 20 r/min, respectively, 20,000×. The direction of *arrows* showed the extrusion direction



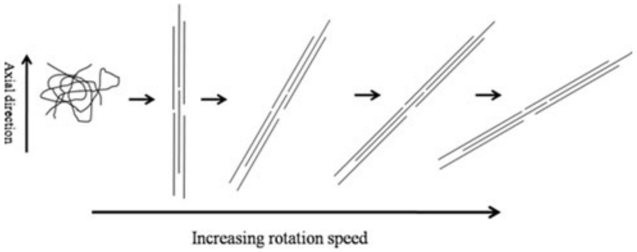


Fig. 6 Schematic illustration of the relation between the orientation state and the rotation speed of die

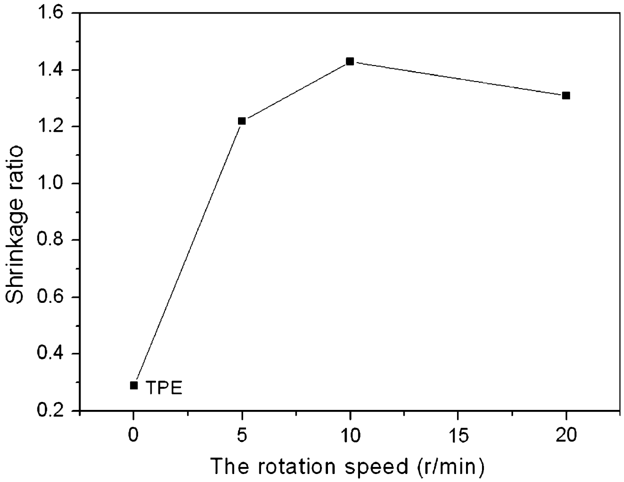


Fig. 7 Shrinkage ratio of the rotation pipes and the conventional pipe along the extrusion direction

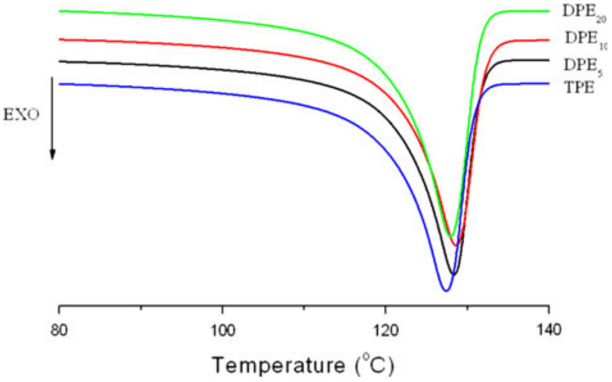


Fig. 8 DSC curves of the PE pipe by the conventional extrusion and the pipes obtained at the different die rotation speed

orientation degree was likely to go up. Just as shown in the results of SEM, the oriented molecules in the rotational pipe were most dense, compact and highly aligned. It was well known that the oriented molecular chains were of a more thermostatically stable structure. Consequently, the melting temperature of the samples was enhanced by the die rotation. However, when the die rotated, a factor of the hoop-shear friction must fall into our consideration. It had been proved that when the polymer melt was under the effect of the strong shear stress, a large amount of heat may be generated to affect the polymer's crystallization and have the molecular chains relax [30]. Thus, high rotation speed could weaken the orientation effect induced by the die rotation so that the melting temperature decreased.

The relationship between rotation speed, morphological structure, and slow crack growth resistance

The resistance to SCG is primarily determined by two factors: the yield stress of the matrix and the disentanglement rate of the fibrils at the base of the craze [31]. According to the extensive research of Brown and his co-workers [11, 28], there were more molecules that bore the applied stress in the fibrils when the orientation direction of molecular chains was parallel to the applied stress than that when the original structure was perpendicular to the applied stress, and therefore the average force per molecule might decrease. Consequently, the disentanglement rate of the fibrils decreased and the resistance to SCG increased with the increasing orientation degree. The internally pressurized pipe was subjected to a hoop stress twice as high as the axial stress in its application. However, in the conventional extrusion process of plastic pipes, the molecular chains were oriented along the axial direction, which was disadvantageous to the resistance to SCG. The orientation of the molecular chains along the hoop direction was expected to improve the pipe's resistance to SCG. During the rotation extrusion, a hoop stress imposed on the polymer melt led to the molecular orientation deviate from the axial direction so as to induce the multi-axial orientation of molecular chains and increase the orientation of the molecular chains along the hoop direction; moreover, a high rotation speed could enhance the hoop stress and have the molecular chains orientation at the bigger angles relative to the axial direction. Therefore, the number of the oriented molecule along the hoop direction was more than that by the conventional extrusion. As a result, the resistance to SCG was perfected and more improvement could be achieved with the increasing rotation speed. However, high rotation speed may generate a large amount of heat to weaken the orientation effect induced by the die rotation. Thus, the resistance to SCG decreased.

Conclusion

In this article, a novel rotation extrusion processing system was adopted to extrude the HDPE pipe. This rotation extrusion could exert the hoop stress on the polymer melt to lead to the molecular orientation deviate from the axial direction so as to induce the multi-axial orientation of molecular chains and increase the orientation of the molecular chains along the hoop direction. As a result, the hoop orientation degree in the PE pipe increased and the resistance to SCG was enhanced greatly. Compared to the PE pipe produced by the conventional extrusion, the crack initiation time of the PE pipe manufactured by the novel method increased from 27 to 57 h. However, high rotation speed may generate a large amount of heat to weaken the orientation effect induced by the die rotation, and decrease the resistance to SCG.

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